IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN THE APPLICATION OF:

MATTHEW R. SIVIK.

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SERIAL NO.: 10/806,591 EXAMINER: AMY T. LANG

FILED: MARCH 23, 2004 GROUP ART UNIT: 1714

TITLE: FUNCTIONALISED POLYMER COMPOSIITON FOR GREASE

Wickliffe, Ohio

Declaration Under Rule 1.132

Sir,

I, Dr Gareth Fish hereby declare:

I obtained a BSc (Hons) in Chemistry in 1984 and a Ph.D. in Mechanical Engineering (Tribology) in 1990 from the Imperial College of Science, Technology and Medicine, University of London. My Ph.D. was in the field of the lubricating properties of fire resistant lubricants. From August 1990 to May 2007, I was employed by GKN plc as a grease specialist rising from senior, then principal technologist to assuming the role global grease technology manager in June 1999, initially in England and from August 2002 in Auburn Hills, Michigan. I have been employed by The Lubrizol Corporation since June 2007 as a Technology Manager for grease. I have a total 25 years experience in the field of lubricants and grease. Since 1990 I have specialized in the field of grease.

I have written nineteen technical papers on the subject of greases and have been granted two patents (see Annex for list of publications). I have also taught classes in England, the

USA and abroad on lubricating greases since 1991. In 2002 I was invited to teach a class on Automotive greases to the NLGI (formerly the National Lubricating Grease Institute, the foremost trade body for Greases in the USA and internationally), "Basic Grease Course." I have taught this class since on numerous occasions and in 2007 I was invited to take over the running of the Basic Grease Course as co-chair of the NLGI education Sub-committee and I continue to run this grease course on behalf of the NLGI.

I am an NLGI certified Lubricating Grease Specialist. This required passing a 100 question written NLGI examination, answering questions on my knowledge of greases. My passing score was >95%. I am also a Society of Tribologists and Lubrication Engineers (STLE) Certified Lubrication Specialist (CLS)
A copy of my Publications and bibliography is also attached.

I confirm that I have read the application of the present invention and cited prior art references Olson (US 5,308,514) and Smith (US 4,966,722) that were listed in the office action mailed 19 May, 2009.

The subject matter of the application by Sivik et al is that of calcium sulphonate greases. As described in the application and previous office action responses, the independent claim of Sivik relates to a calcium sulphonate grease which has special and unique properties over the nearest prior art (Olson).

To demonstrate that the Sivik invention has unexpectedly better results than Olson, I prepared four calcium sulphonate greases as are summarised in the table below (G1 to G4). The table also summarizes the characterization and test data for each grease.

Grease 1 (G1) is a calcium sulphonate grease containing succinic acid. This grease is representative of the teachings of Olson. Grease 2 (G2) is a calcium sulphonate grease containing dodecyl succinic acid. Grease 3 (G3) is a calcium sulphonate grease containing a C_{20}-alkyl succinic acid. Grease 4 (G4) is a calcium sulphonate grease containing a mixed alkyl succinic acid. The succinic acid is a mixture of C_{16-36}-alkyl

succinic acids. Greases G2 to G4 are representative of the Sivik invention. The greases were prepared as shown in the following table:

	Comparative Grease	Inventive Greases		
	G1	G2	G3	G4
Weight of 400 TBN overbased sulphonate (g)	700	700	700	700
Weight of sulphonate in mixture (g)	129.5	129.5	129.5	129.5
Mole Equivalents of the succinic acid added	0.14	0.14	0.14	0.14
Weight of succinic acid added (g)	16.50	40.00	55.80	57.26
Added heavy paraffinic bright stock oil (g)	350	350	350	350
propan-2-ol promoter	80	80	80	80
water	70	70	70	70
wt % oil added	33.15	32.34	31.87	31.83
wt % oil in final grease	60.82	59.70	58.64	58.56
wt % calcium sulphonate in final grease	39.18	40.50	41.36	41.44

As is noted in the table above, the mole equivalents of each grease G1 to G4 has the same mole equivalent of the named succinic acid. Therefore G1 to G4 are proper side by side comparison of inventive and comparative examples based on mole equivalents.

After gelling and stripping to remove the excess water and promoters the visual appearance, penetration, and dropping point of the grease were determined. The greases were characterised as follows:

	Comparative Grease	Inventive Greases		
	G1	G2	G3	G4
Unworked penetration	238	263	338	308
Worked penetration	241	272	338	316
Color	Greenish Brown	brown	brown	brown
Appearance	Stiff and tacky with lumps	Clear and tacky	Clear, Soft and tacky	Clear, Soft and tacky
Alkalinity (mg/g KOH eq)	15.21	13.8	13.19	13.66
Water spray off (D4049)	6.3	3.7	2.6	5.0

The comparative grease G1 formed with the succinic acid was a stiff dark green opaque color, whilst the inventive greases using the hydrocarbyl substituted acids formed clear dark brown greases.

Greases G1 to G4 are evaluated for water spray-off properties by the methodology of ASTM D4049. The results obtained were:

	Comparative Grease	Inventive Greases		
	G1	G2	G3	G4
Water spray off (D4049)	6.3	3.7	2.6	5.0

The above table shows that the water spray-off of inventive greases G2 to G4 are better than comparative grease G1. In the field of greases it is normal to compare greases with the same or substantially the same worked penetrations. The comparative grease with the succinic acid (G1) is very much stiffer than the other three inventive greases (G2 to G4). Consequently, a person of ordinary skill in the art of preparing and characterizing grease would not consider a valid comparison to compare G1 with inventive greases G2 to G4.

Even with this in mind, the result clearly show that the compositions of the present invention (G2-G4) are significantly different as to water spray-off than that representative

of the reference Olson (G1). The inventive samples all provide better to improved water spray off properties.

In order to provide another comparison of a calcium sulphonate grease containing (i) succinic acid (G1) and a grease containing (ii) dodecyl succinic acid (G2), two greases were prepared from the G1 and G2 bases as above, with the aim of having a similar penetration range, in other words, the small differences in formulation between these new samples are solely to ensure their penetration range is similar, thus providing a comparison of greases formulated to have similar performance, where the only difference is in the additive of interest. These re-balanced greases are G5 (representative of Olson) and G6 (inventive). The greases were characterised and evaluated for water spray-off performance using the same test methods described above, with the results shown in the following table:

	G5 (Comparative with succinic acid)	G6 (Inventive with dodecyl succinic acid)
Weight of grease	540	540
Additional oil (g)	90	90
Weight of the succinic acid	8.42	19.95
Mole equivalents of the succinic acid	0.07	0.07
% thickener in final grease	33.58%	34.72%
% oil in final grease	66.42%	65.28%
Unworked penetration	303	306
Worked penetration	297	303
Color	Greenish Brown	brown
Appearance	Soft and tacky with lumps	Soft, clear and tacky
Water spray off (D4049)	15.7	9.6

The water spray-off data demonstrates that when comparing calcium sulphonate greases with similar penetration values, grease with the comparative grease (G5) with succinic

acid has inferior water spray-off performance compared to inventive grease (G6) containing dodecyl succinic acid. This unexpected result shows the benefits of the present invention.

Overall the results indicate that a calcium sulphonate grease containing a non-polymeric hydrocarbyl substituted derivative of succinic acid, wherein the hydrocarbyl contains about 4 to about 40 carbon atoms has improved water spray-off properties than a similar calcium sulphonate grease. This unexpected improvement is seen when the grease formulations are identical but for the presence of the additive of interest, and also when the grease formulations have been balanced to provide similar penetration ranges.

The Examiner has used the prior art disclosure of Smith to provide a teaching of succinic acids that can be combined with the calcium sulphonate grease of the Olson reference. Olson relates to calcium sulphonate greases. Smith relates to the field of lubricants for internal combustion crankcases. Lubricants for internal combustion crankcases do not contain a grease thickener. This means that a lubricants for internal combustion crankcases generally are amorphous liquids without structure. The calcium sulphonate detergents used in crankcase oils have amorphous calcium carbonate present i.e. no crystalline structure. In contrast, a calcium sulphonate grease employs calcium sulphonate as a grease thickener. The grease thickener provides the structure which retains oil within the matrix of the grease. The calcium carbonate form used in grease is crystalline calcite or vaterite or a mixture of these two crystalline forms (see Olson table II column 10 line 10).

The differences in structure noted above between internal combustion engine lubricants and calcium sulphonate grease significantly distinguishes the technology of grease from that of crankcase oils for internal combustion engine lubrication.

If a grease thickener were added to lubricants for internal combustion crankcases the result would be a lubricant that is unsuitable to lubricate an engine crankcase because of the differences noted above in structure.

In addition, to the structural differences noted above, Smith employs the succinic acids disclosed therein to reduce haze in the internal combustion engine lubricants. Reducing haze is not a feature associated with greases. Furthermore water spray-off properties are particular to greases and are not relevant to crankcase lubricants. Thus the grease compositions of Olson would not be combined with Smith crankcase lubricants or the additives therein into the grease compositions of Olson.

I further declare that all statements herein made of my own knowledge are true and all statements herein made on information and belief are believed to be true. I understand that willful false statements and the like are punishable by fine or imprisonment or both (18 U.S.C. 1001) and may jeopardize the validity of the application or any patent issuing thereon.

Dr. Gareth Fish

16 NOVEMBER 2009

Date

Bibliography

2 Granted Patents

GB 2,287,715 Fish, G "Greases"

US 6,656,890 Fish, G et al "Grease composition for Constant Velocity joints" (with T.D. Davies, J.S. Cooper, J.E and R.G. Isaac)

Published Papers

1989 I. Mech. E. – (with H.A. Spikes) "The Fatigue Lives of Water-based Hydraulic Fluids in Rolling Contact" presented at Advances in Water Based Hydraulics an I. Mech. E. Tribology Group Seminar.

1990 I. Mech. E. – "Comparison of Laboratory Scuffing load Tests" presented at Load Carrying Capacity Testing of Lubricants, an I. Mech. E. Aerospace Industries Division Seminar, London, England, May 23rd 1990.

1994 I. Mech. E. – (with P. Ratcliffe of Fuchs (UK) Lubricants) – "CV Joint Lubrication From the 50's to the Future" presented at Recent Advances in Driveline Technology, an I. Mech. E. Tribology Group Seminar, December 1994.

1995 I. Mech. E. Autotech 95 "Development of Advanced Constant Velocity Joint Lubricants" published as Driveline - Autotech 95 Seminar 24 and in Automotive Powertrains I. Mech. E. Seminar Publication 1996-8 MEP ISBN: 1 86058 020 3 p227-235.

1997 I. Mech. E. Autotech 97 (with S.J. Cole) "Tribology and Lubrication of Constant Velocity Joints" Automotive Engines and Powertrains I. Mech. E. Seminar Publication 1997-6 MEP ISBN: 1 86058 114 5 p97-106.

1997 European Lubricating Grease Institute (ELGI) Annual Meeting in Berlin "Constant Velocity Joint Greases" Published in ELGI Eurogrease.

1997 World Tribology Congress (with S.J. Cole) "The tribology of Constant Velocity Ball Joints" MEP ISBN: 1 86058 109 9 p874.

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2001 Additives 2001 – (with Jisheng E) "The Effect of Friction Modifier Additives on Constant Velocity Joint Performance" paper 26 published by the I. Mech. E. March 2001

2001 NLGI – (with Jisheng E) "The Effect of Friction Modifier Additives on CVJ Grease Performance" published as NLGI Spokesman (2002), Vol 66(7) p22.

2006 ELGI published as E, J., Fish, G., Rosenkranz, S. and Reher, F., "Comparison between PDSC and Oxygen Bomb Test Methods for Evaluation of Grease Oxidation Stability", ELGI Eurogrease, 2006 Issue 4, p42-52.

2008 ELGI (with W.C. Ward Jr and Farrukh Qureshi) "Influence of Components Blended to a Target Base Oil Viscosity on Liquid Phase and Lithium Grease Properties" presented at the ELGI 20th Annual Meeting, Lisbon, Portugal April 2008.

2008 NLGI (with W.C. Ward Jr et al) "Extreme Pressure and Tribo-chemical Film Comparisons of Antimony- and Non-Antimony Additive-Containing Lithium Complex Grease" Paper #0809 NLGI 75th Annual Meeting Williamsburg, Virginia, June, 2008 published as NLGI Spokesman (2009), Vol 73(4) p12-21.

2009 ELGI (with W.C. Ward Jr) "Development of Greases with Extended Grease and Bearing Life" presented at the 21st ELGI Annual General Meeting 25th – 28th April 2009 Gothenburg, Sweden.

2009 NLGI (with W.C. Ward Jr) "Development of Greases with Extended Grease and Bearing Life Using Pressure Differential Scanning Calorimetry and Wheel Bearing Life Testing" Paper #0911 Presented at the NLGI 76th Annual Meeting Tucson, Arizona, USA, June 13-16, 2009.

2009 NLGI "Grease and Additive Influences on Fretting Wear" Paper #0905 Presented at the NLGI 76th Annual Meeting Tucson, Arizona, USA, June 13-16, 2009.

2009 World Tribology Congress "Global Grease Industry Trends and Future Technology Needs", (with M. R. Sivik and P. Y Zhu, Presented at the World Tribology Congress 2009, 6 - 11, September, Kyoto, Japan.

Standards, Books and Book Chapters

I was active in re-writing the 2005 SAE J310 "Automotive Lubricating Greases" and issuing the new SAE J2695 "Heavy Duty Vehicle Lubricating Greases" standards for automotive greases.

Chapter on Automotive Greases in ASTM Manual 62 Automotive Lubricants and Testing Handbook (to be published in 2009) (With SJ Nolan).

Re-edited / re-written the Lubrizol Grease Ready Reference Guide in March 2009. This is publication given to Lubrizol customers as a guide to the technology of lubricating greases.